Optimized Method of Sample Preparation and an Automated Examination and Sizing of PZT Ceramic Powders using SEM

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There are multiple forming techniques that are used when engineering ceramics in many areas of technology. Characterizing powders for dimensional stability, surface quality, density and uniformity is very important. The factors described are important for advanced, high-temperature structural parts such as electronic components, impellers, suspensions and complex cutting tools. The preparation, observation and characterization for the electron microscopy of powders to identify material properties that show their characteristics on a micron scale are explained. The ceramic particles used in this case study are PZT powders which are used in electrical applications such as piezoelectric ceramic ultrasound, SONAR, automotive and capacitive devices in multiple industrial applications, but the techniques explained here can be applied to any powder analysis.

Procedures
The use of several techniques is possible however the method explained will use Scanning Electron Microscopy (SEM) in order to achieve accurate Particle Size Analysis (PSA) of bulk powders. Advantages of electron microscopy compared to other PSA techniques are shown in Figure 1. Figure 2 demonstrates the workflow process of preparing powders for automated image acquisition with the optional use of carbon paste or carbon based conductive tape.

Results
Figure 3A and 3B shows one of 390 images that were automatically acquired by the Hitachi SU3500 VPSEM, segmented and measured for area, roundness, diameter-mean, and other variables for a comprehensive statistical result view.

Conclusion
True 2-dimensional imaging for automating the process of particle size analysis is highly effective. The methods of automated electron microscopy and image analysis software deliver practical applications for Q&A and research development for PZT powders and other powder types. Multiple methods are demonstrated in absolute measurement analysis by non-destructive methods. Customizable workflow processes are demonstrated that can be applied to various industries including powder manufacturing. The sample preparation of powders is essential in allowing the Scanning Electron Microscope to extract extremely accurate information in a highly automated sequencing procedure. This method yields results which can identify single particle dimensions such as major & minor diameters, aspect ratio, area, perimeter and mode. Applying this method in this automated manner will result in a highly accurate Particle Size Analysis of any bulk powder.

References:

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**Figure 1.** A chart is shown above depicting the appropriate PSA technique for a given particle size. Electron Microscopy is capable of resolving almost most particles sizes given.

**Figure 2.** Workflow diagram describing and illustrating an optimal method for preparing particles for EM observation.

**Figure 3A.** Segmented data produced by automated image processing. **Figure 3B.** Statistical data derived from 390 images by multiple particles per tile displayed as a line chart and grouped bar chart.